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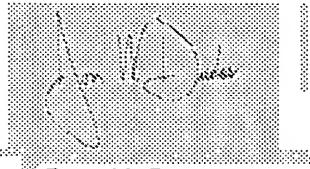
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APPLICATION THAT MET THE REQUIREMENTS TO BE GRANTED A
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Jon W Dudas

Acting Under Secretary of Commerce
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PROVISIONAL APPLICATION FOR PATENT COVER SHEET

This is a request for filing a PROVISIONAL APPLICATION FOR PATENT under 37 CFR §1.53(c).

INVENTOR(S)				
Given Name (first and middle if any)	Family Name or Surname	Residence (City and either State or Foreign Country)		
Brian	Ruby	New York, NY		
Additional inventors are being named on the <u>0</u> separately numbered sheets attached hereto				
TITLE OF THE INVENTION (500 characters max)				
Carbon Nanotube AFM Tips				
CORRESPONDENCE ADDRESS				
Direct all correspondence to:				
[X] Customer Number: 26211				
OR				
[] Firm or Individual Name				
Address				
Address				
City	State	ZIP		
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ENCLOSED APPLICATION PARTS (check all that apply)				
[X] Specification	Number of Pages	11	<input type="checkbox"/> CD(s), Number	
[X] Drawing(s)	Number of Sheets	5	<input type="checkbox"/> Other (specify)	
<input type="checkbox"/> Application Data Sheet. See 37 CFR 1.76.				
METHOD OF PAYMENT OF FILING FEES FOR THIS PROVISIONAL APPLICATION FOR PATENT				
<input type="checkbox"/> Applicant Claims small entity status. See 37 CFR 1.27. <input type="checkbox"/> A check or money order is enclosed to cover the filing fees. <input type="checkbox"/> The Director is hereby authorized to charge filing fees or credit any overpayment to Deposit Account Number: <input type="checkbox"/> Payment by credit card. Form PTO-2038 is attached.			FILING FEE AMOUNT (\$)	
			06-1050	\$80
The invention was made by an agency of the United States Government or under a contract with an agency of the United States Government.				
<input type="checkbox"/> No. <input type="checkbox"/> Yes, the name of the U.S. Government agency and the Government contract number are:				

Respectfully submitted,

Signature Richard P. FerraraDate November 6, 2003Typed Name Richard P. Ferrara, Reg. No. 30,632Telephone No. (212) 765-5070Docket No. 16395-002P01

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**APPLICATION FOR
UNITED STATES PATENT**

in the name of

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Carbon Nanoprobes, LLC

for

Carbon Nanotube AFM Tips

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Carbon Nanotube AFM Tips

TECHNICAL FIELD

This invention relates to nanotubes, and more particularly to carbon nanotube AFM tips.

BACKGROUND

5 Carbon nanotubes are carbon-based molecular structures, which can have diameters of a few nanometers and lengths of several nanometers to several micrometers. Carbon nanotubes have unique mechanical and electrical properties that make them useful for a variety of applications. For example, a carbon nanotube can be used as a probe tip in atomic force microscopy (AFM).

10 Atomic force microscopes use a probe tip to obtain images from a sample by dragging the probe tip over the surface of the specimen or within close proximity to the surface of the specimen. The probe tip that is attached to a cantilever is dragged across the surface of the specimen and the displacement of the cantilever is recorded as the probe tip tracks the topography of the specimen. For example, a laser beam can be shined on the back 15 surface of the cantilever, and the movement of the cantilever can be deduced from changes in the angle of a beam reflected from the surface. The resolution of images produced by AFM is related to the size and shape of the probe tip, and tips with small diameters and high aspect ratios of their length to their diameter can be used to resolve small lateral and vertical features. Carbon nanotubes are useful as AFM probe tips because they are extremely strong 20 and stiff, and can be made of various lengths and aspect ratios to suit the user's needs.

Carbon nanotubes have been produced for use as AFM tips, as described, for example, by Dai, et al. in U.S. Patent 6,346,189 and in U.S. Patent Application Publication US2002/0178846. However, it has been difficult to mass produce such carbon nanotube tips.

SUMMARY

25 In a first general aspect, a method of producing an apparatus with a carbon nanotube tip includes providing a first substrate having a surface with a plurality of precursor tips, growing a nanotube tip on each of the precursor tips, and limiting the growth of each of the nanotube tips to a predetermined maximum length.

In a second general aspect, a method of producing a plurality of apparatuses having nanotube tips includes providing a first substrate having a plurality of precursor tips, growing a nanotube tip on each of the precursor tips, providing a second substrate having a second surface facing the surface, growing a nanotube tip on each of the precursor tips, applying an electrical potential between the second surface and ends of the nanotube tips that are distal from the precursor tips, and separating portions of the substrate having a precursor tip and a nanotube tip grown on the precursor tip.

In a third general aspect, a method of producing an apparatus with a carbon nanotube AFM tip includes providing a first substrate having a surface with a plurality of cantilevers, each cantilever having a catalyst island, growing a nanotube tip on each of the catalyst islands, and limiting the growth of each of the nanotube tips to a predetermined maximum length.

The method can include one or more of the following features. The nanotube tips can be shortened to a desired length. A second substrate having a second surface can be provided at a distance from and facing the first surface of the first substrate. The distance between the second surface and the first surface can be constant and can be between about 5 nanometers and about 5 micrometers, between about 50 nanometers and about 1 micrometer or between about 1 micrometer and about 5 micrometers. The distance between the second surface and the first surface can vary. The second substrate can be conductive. An electrical potential can be applied between the second surface and ends of the nanotubes that are distal to the precursor tips. Each nanotube tip can be separated from other nanotube tips. The nanotube tips can be grown on AFM cantilevers.

Unless otherwise defined, all technical and scientific terms used herein have the same meaning as commonly understood by one of ordinary skill in the art to which this invention belongs. Although methods and materials similar or equivalent to those described herein can be used in the practice or testing of the present invention, suitable methods and materials are described below. All publications, patent applications, patents, and other references mentioned herein are incorporated by reference in their entirety. In case of conflict, the present specification, including definitions, will control. In addition, the materials, methods, and examples are illustrative only and not intended to be limiting.

The details of one or more embodiments of the invention are set forth in the accompanying drawings and the description below. Other features, objects, and advantages of the invention will be apparent from the description and drawings, and from the claims.

DESCRIPTION OF DRAWINGS

5 FIG 1 is a top view of a patterned substrate.

FIG 2A is a side view of a portion of the patterned substrate showing an array of precursor tips.

FIG 2B is a side view of a portion of the patterned substrate showing an array of precursor tips with a layer of resist.

10 FIG 2C is a side view of a portion of the patterned substrate showing an array of precursor tips with a layer of catalytic material on top of the layer of resist.

FIG 2D is a side view of a portion of the patterned substrate showing an array of precursor tips with the resist layer removed and catalytic material on the apexes of the precursor tips.

15 FIG 3A is a top view of a flat, doped substrate with perimeter rails.

FIG 3B is a side view of a sandwich of the patterned substrate and the doped substrate.

FIG 4 is a schematic view of a processing furnace arrangement.

20 Like reference symbols in the various drawings indicate like elements.

DETAILED DESCRIPTION

Referring to FIG 1, a commercially available wafer substrate 100 (e.g., available from Nanodevices, Santa Barbara, CA) has a top surface 102 that includes an unpatterned perimeter 106 and a central patterned portion 104 with 375 prefabricated cantilevers with an pyramidal tip on each cantilever. The wafer substrate 100 can be made of silicon, SiN, or other suitable materials for fabricating an array of cantilevers. The patterned wafer substrate 100 can be attached to a thicker substrate to provide support and flatness to the patterned wafer while chemical vapor deposition (CVD) steps are preformed.

30 Referring to FIG 2A, the pyramidal tips 202 can rise about 1-20 microns above the top surface 102 of the substrate 100. On any given wafer substrate 100 the pyramidal tips

202 can have a uniform height to within about 5% of the specified average height of the pyramidal tips 202 on the substrate 100, however, different wafers from the same manufacturer can have pyramidal tips 202 with different average heights. The wafer surface 102 containing the array of pyramidal tips 202 is spin coated with a resist layer 204 to cover 5 the surface 102 at the base of the pyramidal tips 202, as shown in FIG. 2B. A polymethylmethacrylate (PMMA) material is used for the resist layer 204, but other materials can also be used. The resist layer 204 tends to pool around the bases of the pyramidal tips 202, leaving the apexes of the pyramidal tips 202 uncoated by the resist layer. The resist can be applied to the top surface 102 several times to reach a desired thickness of the resist layer 10 204 that covers most of the pyramidal tips 202 but leaves the apexes 206 of the tips exposed. After an adequate amount of resist has been applied to cover all but the apexes 206 of the pyramidal tips 202, the resist is hardened by baking it on a hot plate at 90 °C for one minute.

Next, a catalyst suspension layer 208 is spin coated onto the apexes 206 of the pyramidal tips 202 and on top of the resist layer 204, as shown in FIG. 2C. The catalyst layer 15 208 contains materials that catalyze the growth of nanotubes, for example, Fe(NO₃)₃. A catalyst suspension solution can be created by dissolving 1.00 mmol (0.196 g) Fe(CO)₅, 0.020 mmol (0.053 g) Mo(CO)₆, 0.100 mmol (0.144 g) octanoic acid and 0.100 mmol (0.242 g) bis-2-ethylhexylamine in 5.00 mL octyl ether and refluxing the solution under an N₂ atmosphere for 30 minutes. The formation of Fe-Mo catalytic nanoparticles in this solution 20 is indicated by the solution turning black. Other catalytic materials can also be used.

The catalytic material solution is then spin-coated onto the resist layer 204 and the apexes 206 of the pyramidal tips 202. After the catalyst layer is applied to the apexes 206 of the pyramidal tips 202, the resist layer 204 is removed in a liftoff process by flushing the substrate 100 in acetone or dichloroethene, thus leaving only the apexes 206 of the pyramidal tips 202 coated with a catalytic material 208, as shown in FIG. 2D. Because the pyramidal tips 202 provide a precursor base upon which nanotube tips are grown, the pyramidal tips 25 202 may also be known as precursor tips. The precursor tips 202 need not be pyramidal in shape but can be of any shape that provides a base upon which a catalytic material 208 may be deposited. Although a method of applying catalyst material to the apexes 202 of precursor tips 202 has been described, catalyst material may be applied to localized islands at the end 30 of individual cantilevers by other methods, and precursor tips may not necessary for locating

catalyst material at the ends of the cantilevers. For example, known masking and lithography techniques can be used to deposit small catalyst islands directly onto the ends of the cantilevers without using precursor tips.

Referring to FIG. 3A, a second substrate 300 has an etched top surface 302 with perimeter rails 304 that are higher than a central flat portion 306. The rails 304 can be of equal height or they can be different heights. The rails can be created on a flat, doped silicon substrate 300 by well-known lithography and etching techniques. Referring to FIG. 3B, when the second substrate 300 is placed on top of the patterned wafer 100, the perimeter rails 304 rest on the perimeter 106 of the patterned wafer 100 so that the central flat portion 306 of the second substrate 300 is disposed at a distance, d, from the top surface 102 of the patterned wafer 100 to form a sandwich having a hollow central tunnel between the patterned wafer 100 and the second wafer 300. Gasses can flow through the hollow central tunnel to grow nanotubes on the apexes 206 of the precursor tips 202. In the tunnel, the apexes 206 are located at a distance from the central flat portion 306 of the second substrate 300, where the distance between the central flat portion 306 and the apexes 206 of the precursor tips 202 is equal to the distance, d, minus the height of the precursor tips 202. If the rails 304 are of equal height, this distance is substantially constant across the entire surface of the flat portion 306. If the rails are of different heights, the distance varies across the central flat portion 306. The distance between the apexes 206 of the precursor tips 202 and the surface 306 of the doped substrate 300 determines the maximum length to which nanotubes can grow on the precursor tips 202. This maximum length can be chosen to be between 5 nanometers and 5 micrometers. The second substrate 300 can be made of doped silicon (e.g., doped with 10^{17} boron atoms per cm^3) so that the substrate 300 is conductive. An electrical contact 312 can be disposed on a back surface 310 of the second substrate 300, such that an electrical potential can be applied to the substrate 300. Although the above sandwich of substrates 100 and 300 has been described with the assumption that the precursor tips 202 rise above the level of the surrounding perimeter surface 106, it is also possible that the precursor tips lie below the surrounding perimeter surface 106, in which case the second substrate 300 must be etched to have a central flat portion that protrudes down into the patterned region 104 of the first wafer 100.

After the wafer sandwich is constructed, a carbon-containing gas is flowed through the central tunnel to grow nanotubes on the apexes 206 of the precursor tips 202 through a CVD process. Referring to FIG. 4, the wafer sandwich is positioned in a second furnace 404, H₂ is input from a first gas source 400 at a rate of 400 standard cubic centimeters per minute (SCCM) into a first furnace 402 until the first furnace reaches a temperature of about 500 °C, while H₂ is input from a second gas source 406 at a rate of 400 SCCM into a second furnace 404 until the second furnace reaches a temperature of about 800 – 900 °C. When both furnaces 402, 404 reach their intended temperatures, the gas flow into the first furnace 402 is changed to CO at a rate of 400 SCCM, and the flow of H₂ into the second furnace 404 is increased to 800 SCCM. While CO flows through the central tunnel of the wafer sandwich and over the surface 102 of the patterned wafer, CO is dissociated and carbon nanotubes grow on the catalyst covered apexes 206 of the precursor tips 202. Nanotubes grow on the precursor tips 202 perpendicular to the surface 102 of the wafer until they reach the flat central portion 306 of the second wafer 300. These gas flows are maintained for about 15 minutes and then the entire system is cooled under a flow of H₂. Growth of nanotubes on the catalyst islands at the apexes 202 of the precursor tips progresses until the nanotubes reach their maximum length near to or in contact with the surface 306 of the doped silicon wafer 300. Although a two furnace setup using CO as a source of carbon for nanotube growth, nanotubes can be grown using other methods as well. For example, a single furnace can be used with methane as the source of carbon, as described by Dai in U.S. Patent No. 6,346,189.

After the nanotubes have been grown on the apexes 206 of the precursor tips 202 and the system has been cooled, the second furnace 404 containing the wafer sandwich is flushed with a non-reactive gas (e.g., Ar, He, Xe, Kr, N₂), and a voltage is applied to the doped wafer 300 and ramped from zero volts to a maximum of about 20 – 50 volts. The application of the voltage to the doped wafer 300 breaks any connection between the nanotubes and the surface 306 of the doped wafer 300 and cleaves the ends of the nanotubes that are distal from the precursor tips from the surface of the second substrate. As the voltage is ramped up the nanotubes are shortened. The nanotubes grown on the precursor tips 202 are all shortened at the same time, such that their free ends become located at substantially the same distance from the surface 306 of the doped wafer 300. In addition to cleaving and shortening by the application of a voltage, the nanotube tips can be shortened and cleaved in other ways, for

example, by the application of a liquid or chemical phase chemical, which causes cleaving or shortening of the nanotubes.

After the nanotubes have been shortened, the individual cantilevers on the patterned wafer 100 can be separated from each other and used in individual atomic force microscopes.

5

OTHER EMBODIMENTS

A number of embodiments of the invention have been described. Nevertheless, it will be understood that various modifications may be made. Accordingly, other embodiments are within the scope of the following claims.

WHAT IS CLAIMED IS:

- 1 1. A method of producing an apparatus with a carbon nanotube tip, the method comprising:
2 providing a first substrate having a surface with a plurality of precursor tips;
3 growing a nanotube tip on each of the precursor tips; and
4 limiting the growth of each of the nanotube tips to a predetermined maximum length.
5
- 6 2. The method of claim 1, further comprising shortening each of the nanotube tips to a
7 desired length.
- 1 3. The method of claim 1, further comprising providing a second substrate having a second
2 surface at a distance from and facing the first surface of the first substrate.
- 1 4. The method of claim 3, wherein the distance between the second surface and the first
2 surface is constant.
- 1 5. The method of claim 4, wherein the distance is between about 5 nanometers and about 5
2 micrometers.
- 1 6. The method of claim 4, wherein the distance is between about 50 nanometers and about 1
2 micrometer.
- 1 7. The method of claim 4, wherein the distance is between about 1 micrometer and about 5
2 micrometers.
- 1 8. The method of claim 3, wherein the distance between the second surface and the first
2 surface varies.
- 1 9. The method of claim 3, wherein the second substrate is conductive.
- 1 10. The method of claim 9, further comprising applying an electrical potential between the
2 second surface and ends of the nanotubes that are distal to the precursor tips.

- 1 11. The method of claim 3, further comprising cleaving ends of the nanotubes that are distal
2 to the precursor tips from the second surface of the second substrate.
- 1 12. The method of claim 1, further comprising separating each nanotube tip from other
2 nanotube tips.
- 1 13. The method of claim 1, wherein the nanotube tips are grown on AFM cantilevers.
- 1 14. A method of producing a plurality of apparatuses having nanotube tips, the method
2 comprising:
3 providing a first substrate having a surface having a plurality of precursor tips;
4 providing a second substrate having a second surface facing the surface;
5 growing a nanotube tip on each of the precursor tips;
6 applying an electrical potential between the second surface and ends of the nanotubes
7 that are distal to the precursor tips; and
8 separating portions of the substrate having a precursor tip and a nanotube tip grown
9 on the precursor tip.
- 1 15. A method of producing an apparatus with a carbon nanotube AFM tip, the method
2 comprising:
3 providing a first substrate having a surface with a plurality of cantilevers, each
4 cantilever having a catalyst island;
5 growing a nanotube tip on each of the catalyst islands; and
6 limiting the growth of each of the nanotube tips to a predetermined maximum length.
- 8 16. The method of claim 15, further comprising shortening each of the nanotube tips to a
9 desired length.
- 1 17. The method of claim 15, further comprising providing a second substrate having a second
2 surface at a distance from and facing the first surface of the first substrate.

- 1 18. The method of claim 17, wherein the distance between the second surface and the first
- 2 surface is constant.
- 1 19. The method of claim 18, wherein the distance is between about 5 nanometers and about 5
- 2 micrometers.
- 1 20. The method of claim 18, wherein the distance is between about 50 nanometers and about
- 2 1 micrometer.
- 1 21. The method of claim 18, wherein the distance is between about 1 micrometer and about 5
- 2 micrometers.
- 1 22. The method of claim 17, wherein the distance between the second surface and the first
- 2 surface varies.
- 1 23. The method of claim 17, wherein the second substrate is conductive.
- 1 24. The method of claim 23, further comprising applying an electrical potential between the
- 2 second surface and ends of the nanotubes that are distal to the precursor tips.
- 1 25. The method of claim 17, further comprising cleaving ends of the nanotubes that are distal
- 2 to the precursor tips from the second surface of the second substrate.
- 1 26. The method of claim 15, further comprising separating each carbon nanotube AFM tip
- 2 from other carbon nanotube AFM tips.

1

ABSTRACT

A method of producing an apparatus with a carbon nanotube tip includes providing a first substrate having a surface with a plurality of precursor tips, growing a nanotube tip on each of the precursor tips, and limiting the growth of each of the nanotube tips to a
5 predetermined maximum length.

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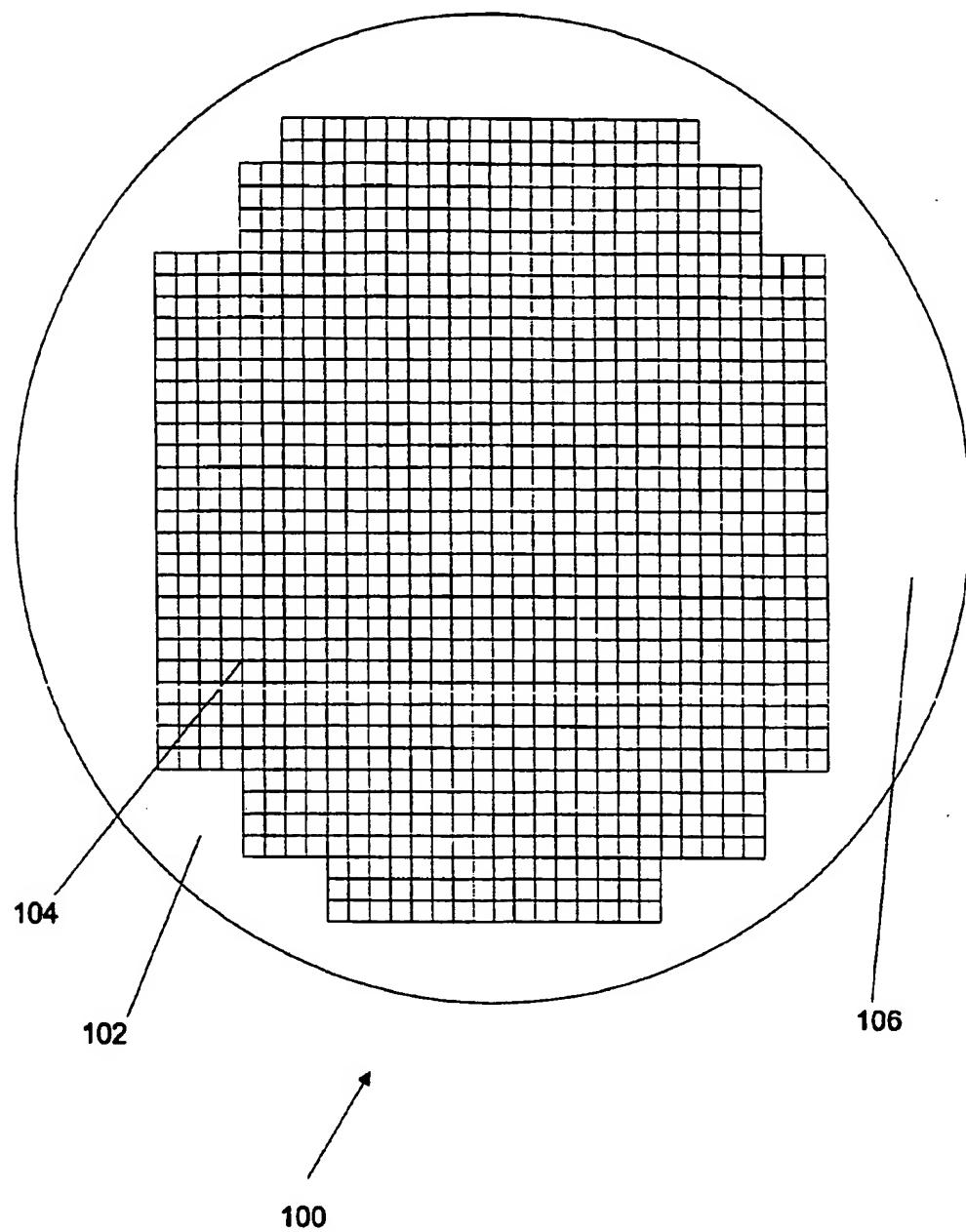
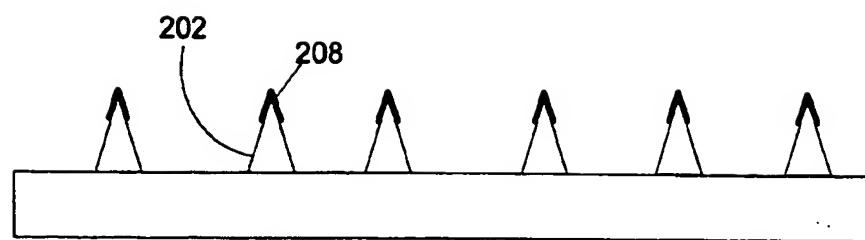
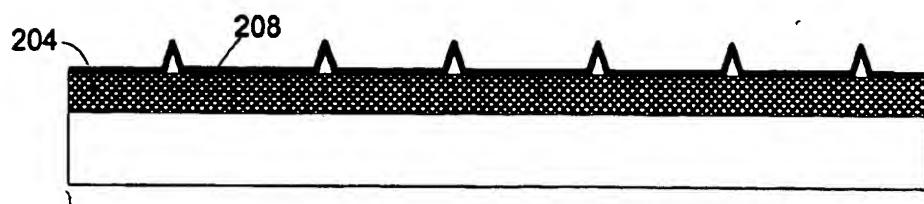
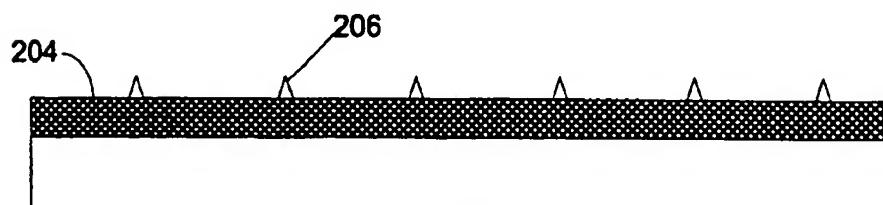
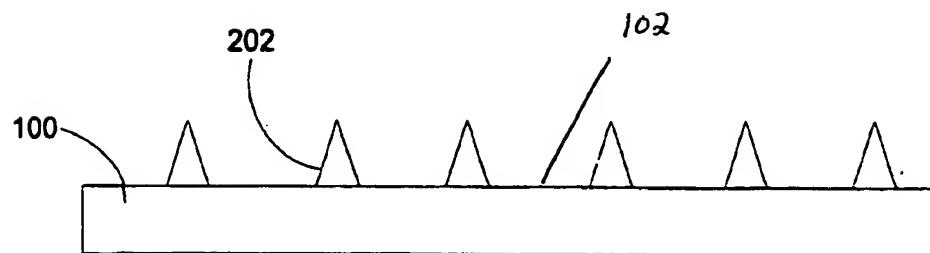


FIG. 1



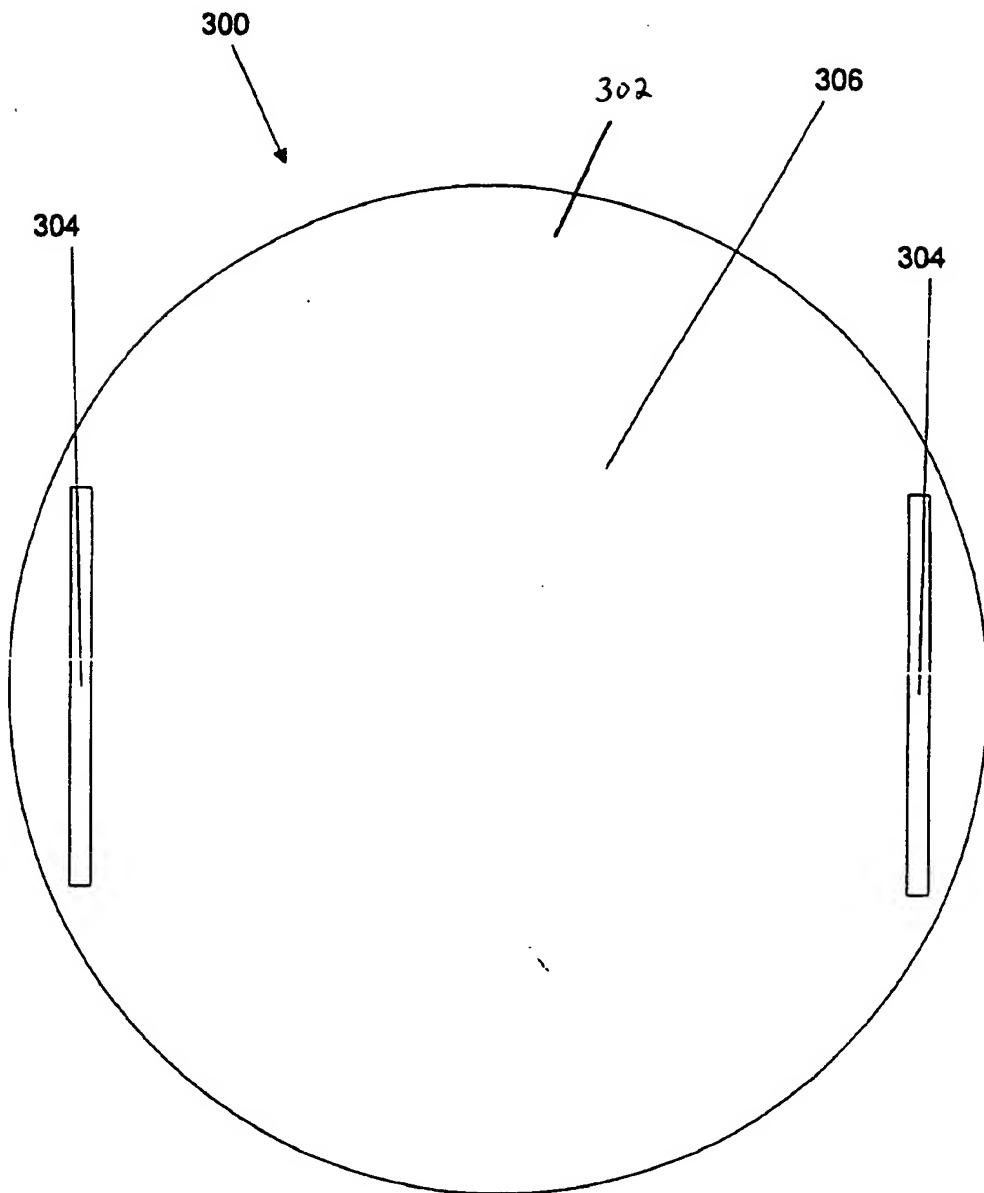


FIG. 3A

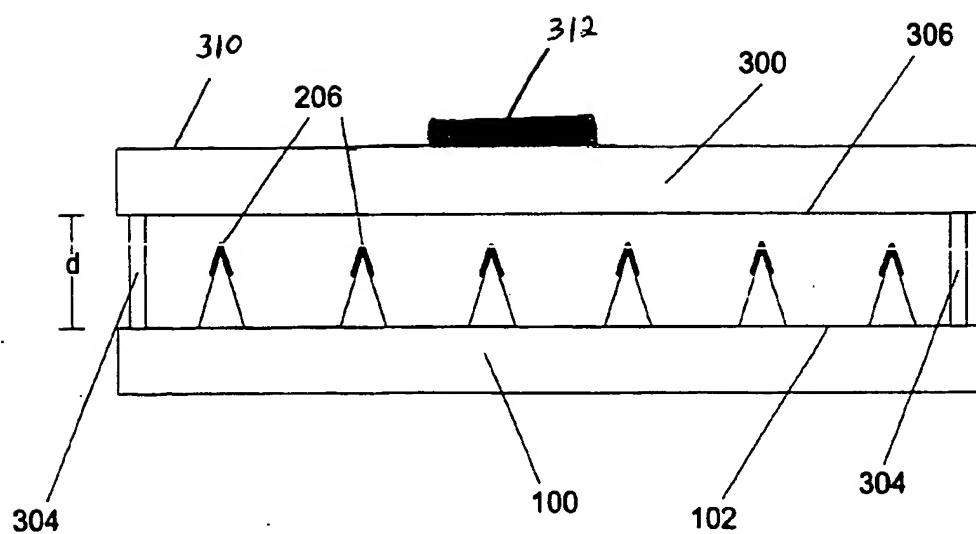


FIG. 3B

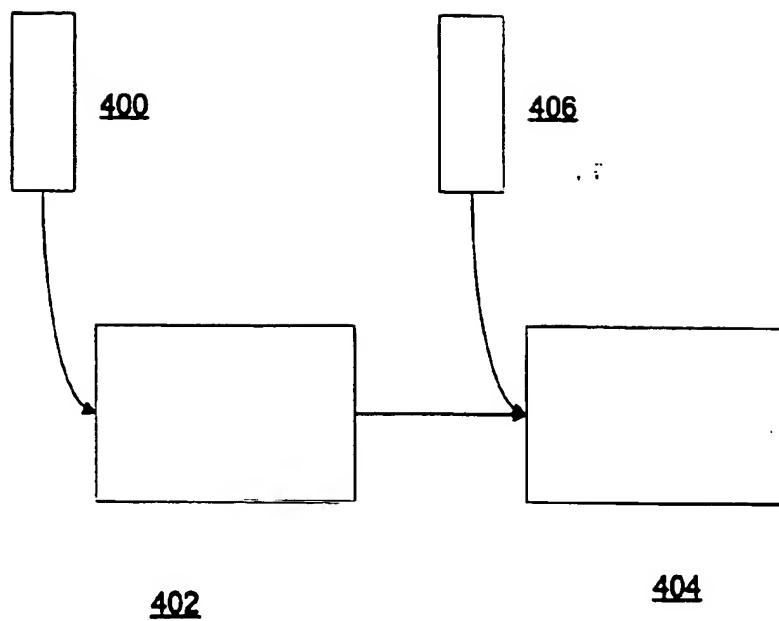


FIG. 4

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